BS 8472:2011

BSI Standards Publication

Methods for the assessment of the oxo-biodegradation of plastics and of the phyto-toxicity of the residues in controlled laboratory conditions

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Contents

Summary of pages

This document comprises a front cover, an inside front cover, pages i to ii, pages 1 to 8, an inside back cover and a back cover.

Foreword

Publishing information

This British Standard is published by BSI and came into effect on 30 June 2011. It was prepared by Technical Committee PKW/0, *Packaging*. A list of organizations represented on this committee can be obtained on request to its secretary.

Relationship with other publications

The following standard is similar but different in scope to this standard.

- ASTM D 6954-04, *Standard Guide for Exposing and Testing Plastics that Degrade in the Environment by a Combination of Oxidation and Biodegradation*.

Presentational conventions

The provisions of this standard are presented in roman (i.e. upright) type. Its methods are expressed as a set of instructions, a description, or in sentences in which the principal auxiliary verb is "shall".

Commentary, explanation and general informative material is presented in smaller italic type, and does not constitute a normative element.

Contractual and legal considerations

This standard does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

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Compliance with a British Standard cannot confer immunity from legal obligations.

0 Introduction

0.1 General

The methods described in this British Standard measure the mineralization of the carbon chains of plastic under controlled laboratory conditions, including the presence of soil micro-organisms and oxygen. The partially mineralized products of the degradation and oxo-biodegradation tests can then be assessed for their effect on seed germination and plant growth.

0.2 Title of the standard

The term "oxo-biodegradation" is defined in **3.8** as it is defined in CEN/TR 15351:2006, **5.2** as "degradation identified as resulting from oxidative and cell-mediated phenomena, either simultaneously or successively".

Oxo-biodegradation is not restricted to man-made polymers. It was first recognized in the biodegradation of natural rubber [1] and it occurs in natural materials such as lignocelluloses, probably mediated by enzymes that produce free radicals [1]. This is analogous to the redox reactions of transition metal ions, where oxygen radicals such as .OH and .OOH are certainly involved in the initiation step. Propagation of the chain reaction occurs primarily on both natural and synthetic products through ROO. This chemistry is readily understood by biochemists since it is widely discussed in both materials and biological chemistry literature (see [1 to 4] and in many text books). Further elaboration in this standard does not seem to be appropriate.

In the environmental exposure of plastics both mechanisms, abiotic and biotic operate together and the micro-organisms rapidly remove the biodegradable oxidation products in a synergistic process. It is difficult and time consuming to reproduce this in the laboratory and for convenience the two processes corresponding to weathering, which is an abiotic process, and biodegradation have to be carried out in separate tests.

ASTM also recognizes the two operations in ASTM D 6954-04. This is oxo-biodegradation, although ASTM does not use this term.

0.3 Use of this standard

This standard defines a specific template to be used for the reporting of results in order to standardize communication and avoid confusion.

This standard is not a specification. Testing according to this standard does not provide any recommendation about the suitability of the tested products for any particular application.

1 Scope

This British Standard describes methods for determining:

a) degradation by oxidation (abiotic tests);

b) biodegradation (biotic test in soil); and

c) phyto-toxicity (plant growth tests);

of plastics materials and products.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ASTM D 5510-94(2001), *Standard Practice for Heat Aging of Oxidatively Degradable Plastics*

BS EN ISO 4892-3, *Plastics – Methods of exposure to laboratory light sources – Fluorescent UV lamps*

BS EN ISO 17556, *Plastics – Determination of the ultimate aerobic biodegradability in soil by measuring the oxygen demand in a respirometer or the amount of carbon dioxide evolved*

OECD 208, *OECD guidelines for the testing of chemicals – Terrestrial plant test: seedling emergence and seedling growth test*

3 Terms and definitions

For the purposes of this British Standard, the following terms and definitions apply.

3.1 abiotic
without the action of living organisms

3.2 biodegradation
degradation of a polymeric item due to cell-mediated phenomena

*NOTE Source: PD CEN/TR 15351:2006, **5.2**.*

3.3 biotic
through the actions of living organisms

3.4 carbonyl index
absorbance of the carbonyl band normalized to an invariant absorbance of the polymer

NOTE See Grassie and Scott [2].

3.5 degradation
change in initial properties due to chemical cleavage of the macromolecules forming a polymeric item, regardless of the mechanism of cleavage

3.6 mineralization (aerobic)
conversion to carbon dioxide, water and other inorganic chemicals

3.7 oxo-degradation
degradation resulting from oxidative cleavage of macromolecules

NOTE 1 Similarly, prefixes like thermo (for the action of heat), photo (for the action of light) are to be used whenever one wants to indicate an identified mechanism of degradation.

*NOTE 2 Source: PD CEN/TR 15351:2006, **5.2**.*

3.8 oxo-biodegradation
degradation resulting from oxidative and cell-mediated phenomena, either simultaneously or successively

NOTE 1 Similarly, prefix like thermo (for the action of heat), photo (for the action of light) are to be used separately or in combination whenever one wants to indicate the involvement of various identified mechanisms of degradation.

*NOTE 2 Source: PD CEN/TR 15351:2006, **5.2**.*

4 Principle

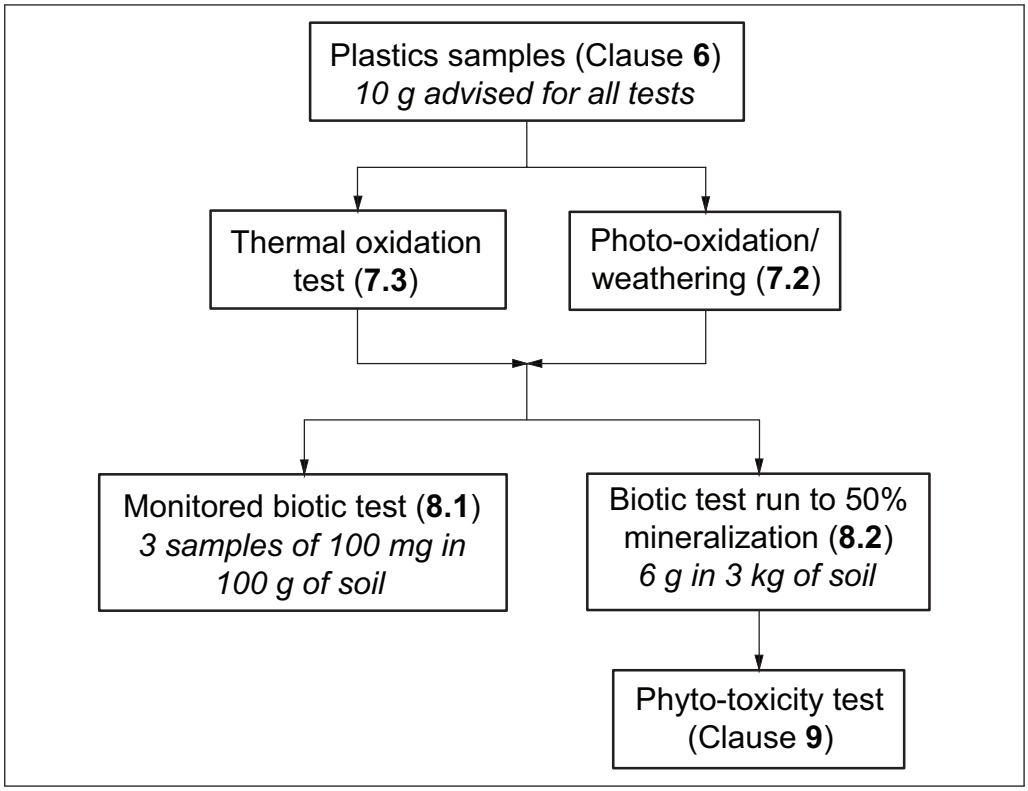
Plastics specimens are subjected to some or all of the following tests (see Figure 1).

a) *An oxidation/abiotic test* (see Clause **7**) by photo-oxidation and/or by thermal oxidation. Degradation to embrittlement is measured by flex test or by friability test.

b) *A biodegradation/biotic test* (see Clause **8**) on residue embrittled by oxidation as per the test in Clause **7**. Mineralization is measured as carbon dioxide evolved as a percentage of the theoretical yield for complete mineralization of total organic carbon content.

c) *A phyto-toxicity/plant growth test* (see Clause **9**) on partially evolved residue from the biodegradation test in Clause **8**. Phyto-toxicity is measured as seed germination and crop biomass compared to control soil.

Figure 1 **Abiotic, biotic and eco-toxicity tests for plastics**



5 Materials and apparatus

NOTE Other equipment is required for referenced test methods.

5.1 *Accelerated weathering device*, typically utilizing a UV lamp 400 W emitting between 290 nm and 450 nm, capable of alternating exposure to dry, light conditions and wet, dark conditions at a minimum ratio of 5:1.

5.2 *Forced-air ventilation oven*, capable of maintaining the internal volume at (50–70) °C ±2 °C, and not exceeding 80 °C in operation, capable of exchanging the unoccupied volume of the oven once per hour, which conforms to ASTM D 5510-94(2001).

6 Sample preparation

6.1 Select requirements for samples either from the product or based on the anticipated product. In particular, specify samples of a given mass that are either:

a) all or part of the product, including the thickest parts; or

b) a sample of the material to be used in a product, at the expected maximum thickness.

6.2 Measure and/or cut out sufficient samples of sufficient size and mass for the selected tests, from a single batch of material.

*NOTE It is recommended that 4 g are prepared for the biodegradation test (**8.1**) and a sample of 6 g is needed for input to the biodegradation process in **8.2** to produce sufficient quantities at 50% mineralization for the phyto-toxicity tests (Clause **9**). That is, 10 g in total.*

6.3 Measure and/or cut out test samples on a clean, dry grease-free surface.

6.4 Record the dimensions and masses of the prepared samples.

7 Oxidation tests

7.1 Introduction

Oxidation (abiotic) testing shall be by photo-oxidation/weathering (**7.2**) and/or by thermal oxidation (**7.3**), using separate samples if both tests are performed.

7.2 Photo-oxidation test

Expose the samples (**6.1**) to artificial weathering in the accelerated weathering device (**5.1**) in general accordance with an exposure cycle in BS EN ISO 4892-3.

7.3 Thermal oxidation test

Expose the samples (**6.1**) in the forced-air ventilation oven (**5.2**) either: at a range of temperatures or at a single temperature between ambient and the temperature at which chemical decomposition becomes significant.

7.4 Measurement of oxidation

At intervals appropriate to the material tested (chosen by trial and error) measure the degree of oxidation by one of the following embrittlement tests of the samples.

a) By bending the sample so that its opposite edges touch and assess whether the sample fractures in a brittle manner.

NOTE 1 A more precise assessment of the degree of oxidation may be obtained by measuring the flexural properties in accordance with BS EN ISO 178.

b) By rubbing the sample between thumb and first finger (friability) and assess whether the sample fragments.

NOTE 2 A more precise assessment of the degree of oxidation may be obtained by measuring the impact-failure energy using a falling dart test in accordance with BS 2782-3.

NOTE 3 Care should be taken to collect sufficient clean material, whether intact or fragmented, for subsequent tests.

NOTE 4 IR spectroscopy of the test residue to find the carbonyl index is a useful method for confirming oxidation.

*NOTE 5 Samples that are not embrittled under **7.4**a) or **7.4**b) may be returned to the test chamber for further exposure.*

7.5 Test termination

Terminate the test and record the time to embrittlement when:

a) samples break under the embrittlement test in **7.4**a); or

b) samples fragment under the embrittlement test in **7.4**b).

8 Biodegradation test

8.1 Test three samples recovered from the oxidation test (Clause **7**) that have reached condition **7.5**a) or **7.5**b) according to the biodegradation (biotic) test in BS EN ISO 17556, with the possibility of terminating the test beyond six months and recording the test duration.

8.2 Obtain samples for the plant growth test by: running the biodegradation process in bulk using at least 6 g of oxidized material in 3 kg of soil (a starting concentration double that recommended in BS EN ISO 17556) and taking the sample as soon as practicable after the point where the monitored test has reached 50% of theoretical mass of carbon dioxide evolved.

NOTE It may be necessary to use a higher concentration of sample to soil than recommended in BS EN ISO 17556. Care has to be taken to maintain other experimental conditions.

9 Phyto-toxicity test

Measure the effect of the partly evolved plastics samples on plant germination number and biomass, in accordance with OECD 208, using 0.5 kg per pot. Use the test substance from **8.2** and use the negative control samples from the biodegradation test. Use at least two plant species from two of the three mentioned categories of OECD 208. Record the germination number and biomass crop yield of plants, compared to the control soil.

10 Test report

A test report containing the following information as a minimum shall be prepared following the template shown in Figure 2.

a) For all tests:

   1) a reference to this standard, i.e. BS 8472:2011;

   2) the material, its constituents and/or its unique manufacturer's identification code; and

   3) initial sample size(s), thickness(es) and mass(es).

b) For photo-oxidation tests:

   1) the type of UV lamp used as categorized in BS EN ISO 4892-3;

   2) the total exposure time and the exposure cycle to which the samples have been subjected; and

   3) the relevant test criteria and results as required in **7.4** and **7.5**.

c) For thermal oxidation tests:

   1) the rate of airflow through the sample/total flow, the temperature due to the forced-air ventilation oven, and the total time of exposure; and

   2) the relevant test criteria and results as required in **7.4** and **7.5**.

d) For biotic tests: the information required for a test report to BS EN ISO 17556.

e) For phyto-toxicity tests: the results required by OECD 208.

Figure 2 **Test report template**

<table>
<tr><td colspan="4">Report on materials test to BS 8472:2011</td></tr>
<tr><td colspan="3">Test laboratory:</td><td>Date of report:</td></tr>
<tr><td colspan="2">Material tested</td><td>Known constituents</td><td>Manufacturer’s ID code</td></tr>
<tr><td>Number of samples</td><td>Sample thicknesses (mm)</td><td>Sample sizes (largest dimension, mm)</td><td>Sample masses (mg)</td></tr>
<tr><td colspan="3">Photo-oxidation</td><td>Tested: Yes/No</td></tr>
<tr><td colspan="2">Lamp used [A)]</td><td>Exposure cycle [A)] and total exposure time</td><td>Test used and results</td></tr>
<tr><td colspan="4">[A)] Type of lamp and exposure cycle selected from BS EN ISO 4892-3.</td></tr>
<tr><td colspan="3">Thermal oxidation</td><td>Tested: Yes/No</td></tr>
<tr><td colspan="2">Rate of airflow</td><td>Temperature and total exposure time</td><td>Test used and results</td></tr>
<tr><td colspan="3">Biotic test, report attached as per BS EN ISO17556 reporting requirements</td><td>Yes/No</td></tr>
<tr><td colspan="3">Duration of test</td><td></td></tr>
<tr><td colspan="3">Phyto-toxicity test, report attached as per OECD 208 reporting requirements</td><td>Yes/No</td></tr>
</table>

Bibliography

Standards publications

For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ASTM D 6954-04, *Standard Guide for Exposing and Testing Plastics that Degrade in the Environment by a Combination of Oxidation and Biodegradation*

BS EN ISO 178, *Plastics – Determination of flexural properties*

BS 2782-3: Method 352F: ISO 7765-2, *Methods of testing plastics – Part 3: Mechanical properties – Method 352F: Determination of impact resistance by the free-falling dart method (instrumented puncture test)*

PD CEN/TR 15351:2006, *Plastics – Guide for vocabulary in the field of degradable and biodegradable polymers and plastic items*

Other publications

[1] Scott G. in *Degradable Polymers: Principles and Applications*, 2nd Edition, ed. Scott G., Kluwer, Chapter 3. 2002.

[2] Grassie, N. and G. Scott., *Polymer degradation and stabilization.* Cambridge Univ. Press, Cambridge. 1985.

[3] Arnaud, Dabin P., Lemaire J., Al-Malaika S., Chohan S., Coker M., Scott G., Fauve A. and Maaroufi M., *Polym. Deg. Stab., 46*, pp 211–224. 1994.

[4] Bonhomme S., Cuer A., Delort A-M., Lemaire J., Sancelme M. and Scott G., *Polym. Deg. Stab.*, **81**, pp 441–452. 2003.

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